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Pulse method for measurement of thermal conductivity of metals and alloys at cryogenic temperatures

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Abstract : A computer controlled experimental facility to measure thermal conductivity of aerospace metals and alloys at cryogenic temperatures using pulse method has been described. The experimental set-up has been calibrated using a standard stainless steel 304 sample The reliability of pulse method has been further confirmed by measuring the thermal conductivity of a copper alloy and an Inconel 718 by both conventional steady state method as well as by pulse technique The agreement between the measured data obtained by both these methods has been found to be within ~3%. Advantages of the present technique have been discussed

Keywords : Thermal conductivity of metals and alloys, pulse technique cryogenic temperatures

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1. Introduction

In condensed matter physics, measurement of thermal conductivity of solids at low temperatures is of great interest because it helps one to identify the type of thermal carriers and their interaction mechanisms [1–4], operative at different temperature zones. Besides, design and development engineers in aerospace industries continue to have urgent need for thermal property data for new alloys and composites. For most materials, specially uncommon alloys and new composites, measured values of thermal conductivity at cryogenic temperatures are not available readily and predictions also can not be made with adequate confidence. Traditionally, heat conductivity experiment is performed using steady-state technique [5–8]. In this method thermal conductivity (λ) is determined by

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noting the steady temperature gradient across the sample for a known quantity of heat when the steady state equilibrium is reached. Steady state method, in spite of being a straight forward one, has the main disadvantage of very long waiting times for both temperature stabilisation, as well as for establishment of a steady state thermal gradient. A typical estimate by Reese [9] for the time (t) taken for the temperature gradient (ΔT) to reach within 1% of its equilibrium value is :

$$t = \frac{18.4}{\pi^2}\frac{C}{K}L^2 \text{ (sec)}, \tag{1}$$

where, C is the specific heat per unit volume. Thus, a stainless steel sample of ~50 mm long would require an equilibrium time of about 1 hour at 20 K. It is important to note that long waiting times also influence precision owing to temporal offset drifts.

Such disadvantages of steady-state method may be eliminated to a large extent by using *Pulse* or, *Non-steady-state* method, in which the bath temperature is allowed to drift slowly. In the present communication, a brief description of the design and the performance of the facility for thermal conductivity measurement at cryogenic temperatures by pulse method is reported. This facility has been developed for the investigation of thermal conductivity of aerospace alloys between 10 and 300 K.

2. Outline of the pulse technique

In pulse method, as the temperature of the bath is allowed to drift slowly and a periodic square wave current (period 2τ) excites the heater, the system never returns to the steady state. Instead, the temperature (T) of the heat source becomes an oscillating function of time. Under such conditions, the thermal conductance (K) of the sample may be expressed in terms of the peak-to-peak amplitude of the signal as [10] :

$$K = \frac{RI_0^2}{(\Delta T)_{pp}} \tanh\left(\frac{K\tau}{C}\right) \tag{2}$$

where, R and I_0 are the heater resistance and the peak current through the heater. τ is the half period of the square wave. Similarly, the heat capacity (C) of the heat source can be expressed as :

$$C = \frac{2RI_0^2}{\left(\frac{\partial \Delta T}{\partial t}\right)_{pp}}\left[1 + e^{-K\tau/C}\right], \tag{3}$$

where $(\delta\Delta T/\delta t)$ is the time derivative of the peak-to-peak amplitude of temperature difference (ΔT). Thermal conductance (K) of the sample can be determined by solving equations (2) and (3) by successive iteration. Thermal conductivity (λ) is then obtained from : $\lambda = K\ (\Delta L/A)$ where A is the area of cross section of the sample and ΔL is the distance over which the temperature gradient is monitored. Influence of error in C on K may be estimated from :

$$\frac{\delta K}{K} = \left[1 - \frac{\sin h\left(\frac{K\tau}{C}\right)}{\frac{K\tau}{C}} \right]^{-1} \frac{\delta C}{C}. \tag{4}$$

It is evident from the above that for $\tau > 4C/K$, a 100% error in C induces an error of less than 1% in K. If $\tau \gg 4C/K$, the system reaches the steady state regime. However, if $\tau \ll 4C/K$, the measured signal does not correlate with the thermal conductance (K).

3. Experimental

The present facility for thermal conductivity measurement between 10 and 300 K has been built using a cryo-refrigerator (APD model 202). It may be noted that the same facility also enables one to measure the thermal conductivity under conventional steady-state conditions, with the difference that for pulse method the heating current is pulsed as square wave with an appropriate time period. At the two ends of the cylindrical sample (length ~40 mm, ϕ ~4 mm) two small copper electrodes were soldered. One of the copper blocks was firmly screwed to the second stage (10 K) of the cryocooler with an indium foil in between in order to ensure excellent thermal contact. The small copper block at the other end of the sample contained a small heater (50 Ω), which was used to generate thermal gradient (ΔT) across the sample. A differential Au + 0.07% Fe *vs* Chromal thermocouple was employed to monitor ΔT across the length (ΔL) of the sample. Absolute temperature (T) of the sample was monitored using a calibrated Si diode sensor (Lakeshore DRT-470). The sample was kept enclosed within a radiation shield (connected to the 10 K stage) and was covered with at least 10 layers of aluminised mylar sheet. The entire assembly was further surrounded by another copper shield thermally anchored to the first stage (40 K) of the cryo-refrigerator. Finally, the sample holder and the radiation shields were enclosed in a stainless steel vacuum shroud. Experiments were performed under a vacuum level of $\sim 1 \times 10^{-6}$ torr so as to make the heat losses due to gas conduction and convection negligible.

Figure 1 shows the schematic diagram of experimental set-up. All voltages were measured by a digital nano voltmeter (Keithley model 181) with a resolution of 10 nV. Pulse heating current of appropriate frequency was generated using a programmable current source (Keithley model 220). The sample temperature was drifted at any desired rate with the help of a programmable temperature controller (Scientific Instruments model 9600). The entire system was interfaced with a PC 386 for continuous data acquisition. Typical time for measurement of thermal conductivity of a stainless steel sample between 10 and 300 K including the cool down time was about eight

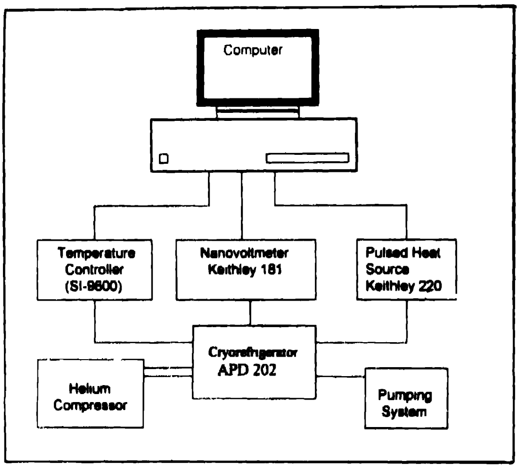

Figure 1. Schematic diagram of the electrical layout and the instrumentation for the measurement of thermal conductivity of metals and alloys at cryogenic temperatures by Pulse method.

hours. Maximum uncertainty estimated in the measurement of thermal conductivity (λ) was ~8%.

4. Results

The facility described above for pulse method of measurement of the thermal conductivity of metals and alloys has been tested successfully with a standard SS-304 sample. A typical

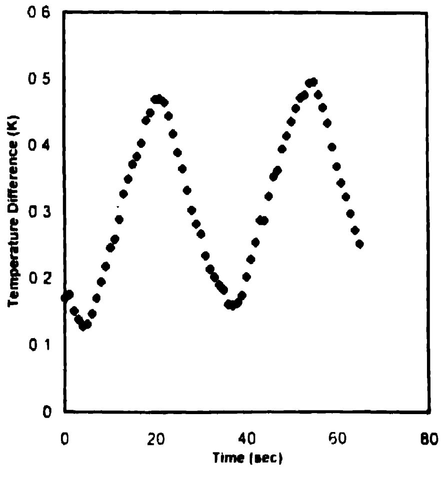

Figure 2. Typical nature of the time dependence of the temperature gradient (ΔT) across the sample observed in Pulse method.

plot for the time variation of ΔT due to square wave current pulse to the sample heater as the bath temperature is slowly drifted is shown in Figure 2. Measured values of λ for

SS-304 sample between 10 and 300 K is shown in Figure 3 along with the NBS data [11]. It may be seen that the agreement between the two is very satisfactory (maximum deviation

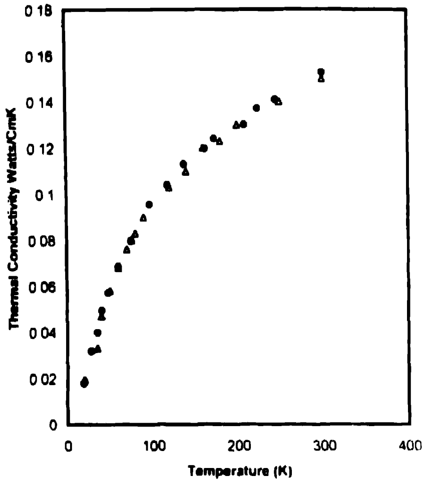

Figure 3. Temperature dependence of thermal conductivity between 15 and 300 K for an SS-304 sample. Series 2 NBS data (Δ) and Series 3 measured by Pulse method (•)

~±5%). The reliability of the pulse method was further established by measuring λ for two more samples (*viz.* Copper alloy and Inconel 718). Thermal conductivity of these two samples between 20 and 300 K obtained by pulse method has been cross checked with the data obtained by conventional steady state method. Figures 4(a)and (b) show the results.

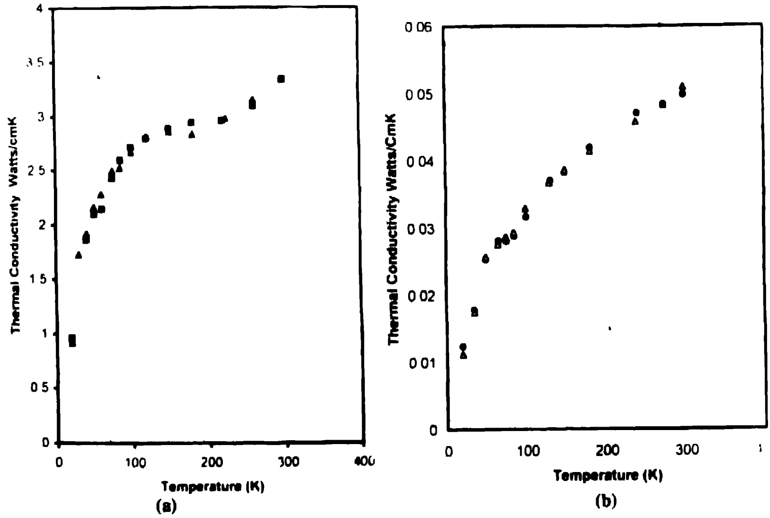

Figure 4. Thermal conductivity as a function of temperature between 15 and 300 K measured by both Pulse method and Steady state method. (a) **Copper alloy** [Series 2 : Pulse method (■) and Series 3 : Steady state method (▲)] and (b) **Inconel 718** [Series 2 : Pulse method (Δ) and Series 3 : Steady state method (•)].

Temperature variation of thermal conductivity between 10 and 300 K for both the samples measured by steady state method and by pulse method agrees to better than ±3%.

As noted earlier, the validity of the pulse method for thermal conductivity measurement depends on the proper choice of the half period (τ) of the square wave pulse applied to the sample heater and the drifting rate of the bath temperature. Principal sources of error in this measurement are associated with the measurement of ΔT, the geometrical factor ($\Delta L / A$) and the time derivative of the peak to peak temperature difference $(\delta \Delta T / \delta t)_{pp}$.

5. Conclusions

An experimental facility for the measurement of thermal conductivity of aerospace metals and alloys between 10 and 300 K using pulse technique has been described. Our test results on various samples show excellent agreement with those measured by conventional steady state method. Pulse method allows faster and accurate measurement of thermal conductivity of metals and alloys with higher point density and hence could be adopted for routine measurements.

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